

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 361 (2009): Normal Butyl Alcohol, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



भारतीय मानक
नार्मल ब्यूटाईल एलकोहल, तकनीकी — विशिष्टि
(तीसरा पुनरीक्षण)

Indian Standard
NORMAL BUTYL ALCOHOL, TECHNICAL —
SPECIFICATION
(*Third Revision*)

ICS 71.100.80

© BIS 2009

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products and Dye Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1953 and subsequently revised in 1962 and 1973. The present revision has been formulated to incorporate the requirement of purity of normal butyl alcohol. Gas chromatographic method has been prescribed for determination of purity, wherein known impurities can also be estimated.

Normal butyl alcohol is used to prepare plasticizers. PVC accounts for the major use of plasticizers. Due to plasticizers, PVC performance improves in respect of flexibility, heat and light stability and facilitates processing using various techniques.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

NORMAL BUTYL ALCOHOL, TECHNICAL — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and test for normal butyl alcohol, technical, which is used as an industrial solvent and especially as a thinner and solvent for paints.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	Title
229 : 1993	Ethyl acetate — Specification (<i>third revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
323 : 2007	Specification for rectified spirit (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
1260 (Part 1) : 1973	Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (<i>first revision</i>)
1448 [P : 18] : 1991	Methods of test for petroleum and its products [P:18] Distillation of petroleum products (<i>second revision</i>)
2362 : 1993	Determination of water by Karl Fischer method — Test method (<i>second revision</i>)
7445 : 1974	Code of safety for acetone

3 REQUIREMENTS

3.1 Description — The material shall be clear, free from sediment and matter in suspension, and shall consist essentially of butan-1-ol ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$).

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in well-closed containers made of galvanized iron or of any other suitable material, with preferably replaceable closures to the provisions of law in force in the country for the time being.

4.1.1 All the containers in which the material is stored shall be clean, dry and leak proof. They shall be kept in a cool place.

4.2 Marking

Each container shall be marked legibly and indelibly with the following information:

- Name of the material;
- Manufacturer's name;
- Net, gross and tare mass;
- Recognized trade-mark, if any; and
- Date of packing.

4.2.1 Each container shall also be marked with the minimum cautionary notice worded as 'FLAMMABLE'.

4.2.1.1 It shall also bear the corresponding symbol for labelling of dangerous goods [see Fig. 5 of IS 1260 (Part 1)].

NOTES

1 Necessary safeguards against the risk arising from storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion (see IS 7445).

2 Except when they are opened for the purpose of cleaning and rendering them free from butyl alcohol vapour, all empty tanks or other containers shall be kept securely closed unless they have been cleaned and freed from butyl alcohol vapour.

Table 1 Requirements of Normal Butyl Alcohol, Technical
(Clause 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			IS No.	Annex
(1)	(2)	(3)	(4)	(5)
i)	Purity, percent by mass, <i>Min</i>	99.5	—	A
ii)	Colour, Hazen, <i>Max</i>	10	—	B
iii)	Relative density at 20°/20°C	0.810 to 0.815	—	C
iv)	Distillation range at 760 mm Hg	Shall distill within the range 116–118°C	IS 1448 [P : 18]	—
v)	Residue on evaporation, g/100ml, <i>Max</i>	0.01	—	D
vi)	Acidity (as CH ₃ COOH), percent by mass, <i>Max</i>	0.015	—	E
vii)	Aldehydes and ketones content (as butyraldehyde, C ₄ H ₇ CHO), percent by mass, <i>Max</i>	0.20	—	F
viii)	Water content, percent by mass, <i>Max</i>	0.10	—	G
ix) ¹⁾	Sulphuric acid test, Hazen units, <i>Max</i>	50	—	H

¹⁾ Applicable when the product is to be used for manufacture of plasticizers.

4.2.2 BIS Certification Marking

The container may also be marked with the Standard Mark

4.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples shall be as prescribed in IS 229.

5.2 Number of Tests

5.2.1 Criteria of Conformity

The lot shall be declared as conforming to the standard if the test results of the composite test sample satisfy the requirements prescribed under 3.2. Otherwise the lot shall be rejected.

5.3 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF PURITY

A-1 GENERAL

This test method is related to determination of the purity of butyl alcohol by gas chromatography and in addition, provides a means for identification of certain known impurities.

A-2 APPARATUS

A-2.1 Gas Chromatograph — Any gas liquid chromatograph equipped with FID detector can be used with the following accessories and operating conditions:

<i>Column</i>	<i>Chromosorb 101</i>
Mesh size	: 80/100
Column length	: 3 m
I.D.	: 3 mm
Column tubing material	: Stainless steel (tubing must be non-reactive with substrate, sample and carrier gas).
Carrier gas	: Nitrogen (Purity — 99.99 mole percent, <i>Min</i>)
Fuel gases	: Hydrogen (30 ml/min) and zero air (300 ml/min)
Syringe	: 10 microlitre
Sample size	: 0.2 microlitre
Electronic integration	: For computation of results integration

A-2.1.1 Instrument Condition

Detector	: FID
Oven temperature	: 180°C
Injector, temperature	: 220°C
Detector, temperature	: 240°C
Carrier gas flow rate	: H ₂ , 30 ml/min

NOTE — The above gas chromatographic conditions are suggestive. However, any GC with different column may be used provided standardization/calibration has been done after setting up chromatographic conditions for the required resolution.

A-2.1.2 Identification, Calibration and Standardization

Install the column in the chromatograph. Set the conditions given above, of column temperature and carrier gas flow that gives the necessary resolution of the components in the sample being analyzed. Set the conditions as such which gives a minimum 10 percent recorder deflection for a 0.1 percent concentration of purity at the most sensitive setting

of instrument. Allow sufficient time for the instrument to reach equilibrium as indicated by stable base line. Adjust carrier gas flow rate to a constant value.

A-2.1.2.1 Identification

Determine the retention time of each component by injecting small amount either separately or in known mixture.

A-2.1.2.2 Calibration and standardization

The response of any given detector varies from one chemical to another. Therefore, calibration is must. Calibration allows the computation of response factor which express the relative response of different components to the detector.

Calibration standard is prepared containing the impurities present in butyl alcohol. During standard preparation the exact amounts of all the components is noted. Inject 0.2 ml of calibration standard in the chromatographic column in accordance with the specified conditions.

Calculate response factor to four decimal places for all components relative to butyl alcohol using following equation:

$$Rf_x = \frac{W_x \times AM}{A_x \times WM}$$

where

Rf_x = response factor for component x relative to butyl alcohol,

W_x = weight of component x, in the calibration standard,

A_x = area of component x, in the calibration run,

AM = area of butyl alcohol in the calibration standard run which is selected as reference component, and

WM = weight of butyl alcohol in the calibration standard.

The component which is unknown in the sample is assigned the relative response factor 1.000 0. It is advisable that the determination of response factors be made on the basis of duplicate analysis.

Response factor should be re-checked after any perceptible change in column or instruments performance.

A-3 PROCEDURE

Inject 0.2 µl of sample into the chromatographic column, using the same conditions as for components identification and standardization. Record and integrate the chromatogram using appropriate attenuation setting that provide optimum peak heights. Measure the area of all the peaks.

A-4 CALCULATION

Calculate the concentrations of sample components using the following equation:

$$C_x, \text{ percent} = \frac{f_x \times R_x}{R_x \times f_x} \times 100$$

where

C_x = component x, weight percent,

R_x = peak response of component x,

f_x = relative response factor of component x, and

$R_x \times f_x$ = sum of the individual component peak responses (R_x) multiplied by their relative response factors (f_x).

A-5 PRECISION AND BIAS**A-5.1 Repeatability**

Two results, should be considered suspect if they differ by more than 0.000 6.

A-5.2 Reproducibility

Two results should be considered suspect if they differ by more than 0.002 7.

ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF COLOUR**B-1 OUTLINE OF THE METHOD**

The colour of the material is compared with that of the colour standard and expressed in terms of Hazen colour units. The Hazen colour, which is defined as the colour of an aqueous solution containing 1 ppm of platinum in the form of chloroplatinic acid and 2 ppm of cobaltous chloride ($\text{CoCl}_2 \cdot \text{H}_2\text{O}$).

B-2 APPARATUS

B-2.1 Nessler Cylinders — two, 100 ml capacity.

B-2.2 One-Mark Graduated Flasks — 250 ml and 500 ml capacities.

B-3 REAGENTS

B-3.1 Cobaltous Chloride Hexahydrate

B-3.2 Hydrochloric Acid — Relative density 1.18 (see IS 265).

B-3.3 Chloroplatinic Acid — Dissolve 250 mg of platinum in a small quantity of aqua regia contained

in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of the hydrochloric acid and again evaporate to dryness. Repeat this operation twice more.

B-3.3.1 Preparation of Colour Standards — Dissolve 0.50 g of the cobaltous chloride hexahydrate and whole of the chloroplatinic acid (see B-3.3) in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500-ml graduated flask. Dilute with water to the mark.

B-3.3.2 Pipette 7.5 ml of this solution (see B-3.3.1) into a 250-ml graduated flask and 25 ml into another 250-ml graduated flask. Dilute with water up to the graduation mark. These diluted solutions are equivalent to 15 and 50 Hazen units respectively and should always be freshly prepared.

B-4 PROCEDURE

Fill one of the Nessler cylinders to the mark with the material to be tested and the other with the colour standard using a white background. Complete the colours.

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF RELATIVE DENSITY

C-1 OUTLINE OF THE METHOD

In this method, mass of equal volumes of material and water are compared.

C-2 APPARATUS

C-2.1 Relative Density Bottle — 25 ml capacity.

C-2.2 Water-Bath — maintained at $27.0 \pm 0.2^\circ\text{C}$.

C-2.3 Thermometer — any convenient thermometer of a suitable range with 0.1 degree or 0.2 degree subdivisions.

C-3 PROCEDURE

Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 27°C . Fill to overflowing by holding the relative density bottle on its side in such a manner as to

prevent entrapment of air bubbles. Insert the stopper and immerse in the water-bath. Keep the entire bulk completely covered with water and hold at that temperature for 30 min. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry and weigh. Again clean and dry the relative bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

C-4 CALCULATION

$$\text{Relative density at } 27^\circ\text{C}/27^\circ\text{C} = \frac{A-B}{C-B}$$

where

A = mass of the relative density bottle with the material, in g;

B = mass of the relative density bottle, in g; and

C = mass of the relative density bottle with water, in g.

ANNEX D

[Table 1, Sl No. (v)]

DETERMINATION OF RESIDUE ON EVAPORATION

D-1 PROCEDURE

From a graduated measuring cylinder, introduce 100 ml of the sample into a 125-ml platinum, silica or borosilicate glass evaporating dish, which has previously been heated to constant mass to a

temperature of 105 to 110°C and cooled in a desiccator. Evaporate the sample to dryness on a hot water-bath and place the dish in an oven at a temperature of $100 \pm 2^\circ\text{C}$. Cool the dish in a desiccator and weigh the residue to the nearest 0.1 mg. This mass is residue on evaporation in g/100 ml.

ANNEX E

[Table 1, Sl No. (vi)]

DETERMINATION OF ACIDITY (AS ACETIC ACID)

E-1 OUTLINE OF THE METHOD

The material is titrated with standard sodium hydroxide solution using phenolphthalein as indicator.

E-2 REAGENTS

E-2.1 Phenolphthalein Indicator — 0.5 percent (m/v). Dissolve 0.5 g of phenolphthalein in 100 ml of

rectified spirit (*see* IS 323) and make it faintly pink by the addition of dilute sodium hydroxide solution.

E-2.2 Standard Sodium Hydroxide Solution — 0.01 N.

E-3 PROCEDURE

Weigh accurately about 100 g of the material into a 500-ml conical flask to the nearest 0.1 g. Pour about

100 ml of freshly boiled and cooled distilled water and add 2 to 3 drops of phenolphthalein indicator. Mix well and note the colour of the solution. A pink colouration indicates the presence of alkalinity. If the solution is colourless, titrate with standard sodium hydroxide solution until a pale pink colour persists for more than 15 s.

E-3.1 Take 100 ml of distilled water in another 500 ml conical flask and carry out blank test.

E-3.2 Calculation

$$\text{Acidity (as acetic acid), percent by mass} = \frac{(V_1 - V_2) \times N \times 0.06}{M} \times 100$$

where

V_1 = volume of standard sodium hydroxide solution used in the test with the material, in ml;

V_2 = volume of standard sodium hydroxide solution used in blank test, in ml;

N = normality of standard sodium hydroxide solution; and

M = mass of the material taken for the test, in g.

ANNEX F

[Table 1, Sl No. (vii)]

DETERMINATION OF ALDEHYDES AND KETONES (AS BUTYRALDEHYDE)

F-1 OUTLINE OF THE METHOD

Hydrochloric acid is liberated by the reaction between hydroxyammonium chloride and any aldehydes or ketones present, and this acid is titrated with sodium hydroxide solution using a standard pH meter (standardized with buffer solutions) with glass-calomel electrodes to determine the end product.

F-2 REAGENTS

F-2.1 Standard Sodium Hydroxide Solution — 0.1 N.

F-2.2 Hydroxyammonium Chloride Reagent — Dissolve 4 g of hydroxyammonium chloride in 20 ml of water, dilute to 200 ml with 95 percent (v/v) ethanol, heat on a water-bath for 30 min and cool.

F-2.3 Carbonyl-Free-Ethanol — Reflux 500 ml of 95 percent (v/v) ethanol (Industrial methylated spirit is not suitable) with 5 g of 2, 4-dinitrophenylhydrazine and 5 drops of concentrated hydrochloric acid (r.d. = 1.18) for 2 to 3 h. Distil off the ethanol slowly using a Widmer or other suitable distillation column of size 300 × 25 mm. Reject the first 50 ml

and collect the next 400 ml distillate, rejecting the remainder. If, in spite of the precautions taken, the distillate is found to be coloured, then it should be redistilled.

F-3 PROCEDURE

Measure 25 ml of the sample into a 250-ml conical flask, add 25 ml of the hydroxyammonium chloride reagent and 50 ml of water. Stopper loosely and heat on a boiling water-bath for 10 min. Cool and titrate with the standard sodium hydroxide solution until end point is reached when pH is 3.8 in the pH meter.

F-4 CALCULATION

$$\begin{aligned} \text{Aldehydes and ketones} \\ \text{(as butyraldehyde, C}_3\text{H}_7\text{CHO), percent by mass} \end{aligned} = \frac{0.0288V}{d}$$

where

V = volume of standard sodium hydroxide solution used; in ml; and

d = relative density of the sample.

ANNEX G

[Table 1, Sl No. (viii)]

METHOD FOR DETERMINATION OF WATER CONTENT

G-1 OUTLINE OF THE METHOD

The water content is determined by Karl Fischer method.

G-2 PROCEDURE

Take about 10 g of the sample, weigh accurately and determine the water content by the procedure given in IS 2362.

ANNEX H

[Table 1, Sl No. (ix)]

SULPHURIC ACID TEST**H-1 PRINCIPLE**

The sample is treated with concentrated sulphuric acid and the developed colour is compared with an agreed colour standard (*see B-3.3.2*).

H-2 APPARATUS

H-2.1 Round Bottom Flask — 250 ml capacity with ground glass stopper.

H-2.2 Burette — 10 ml adjusted to deliver 5 ml of the sulphuric acid at ambient temperature at the rate of two drops per second.

H-3 REAGENTS

H-3.1 Sulphuric Acid — 96 percent (*m/m*).

H-4 PROCEDURE

H-4.1 Clean the round bottomed flask carefully by rinsing it first with sulphuric acid and then with water, and drying it with ethanol.

H-4.2 Rinse it with the test sample and drain well. Measure 75 ml of the test sample into the flask and cool for exactly 5 min in an ice water-bath.

H-4.3 Keeping the flask immersed in the ice water-bath and shaking it continuously, add rapidly from the burette 5.0 ml of sulphuric acid, taking care however that the temperature in the flask does not rise above 20 °C. Stopper the flask and maintain it in the ice water-bath for a total of exactly 3 min from the time of the end of the addition of acid.

H-4.4 At the end of this time, fit a reflux condense to the flask and transfer quickly to a boiling water-bath. Allow the flask to remain on the water-bath for 3 h.

H-4.5 Cool the material in the flask and compare its colour with the colour standard given under **B-3.3.2**.

H-5 REPORTING

Report the colour obtained from the sample as greater than or equal to or less than that of the agreed colour standard (*see B-3.3.2*).

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc: No. PCD 9 (2113).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Telegrams: Manaksanstha
(Common to all offices)

Regional Offices :

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 2323 7617 2323 3841
Eastern : 1/14 C. I. T. Scheme VI M, V. I. P. Road, Kankurgachi KOLKATA 700054	{ 2337 4899, 2337 8561 2337 8626, 2337 9120
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	{ 260 3843 260 9285
Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600113	{ 2254 1216, 2254 1442 2254 2519, 2254 2315
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	{ 2832 9295, 2832 7858 2832 7891, 2832 78792

Branches: AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD.
GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR.
PARWANOO. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.